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Indian Standard
SPECIFICATION FOR
HYDROXYLAMINE SULPHATE,
PHOTOGRAPHIC GRADE

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SPECIFICATION FOR HYDROXYLAMINE SULPHATE, PHOTOGRAPHIC GRADE

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Indian Standard
**SPECIFICATION FOR
HYDROXYLAMINE SULPHATE,
PHOTOGRAPHIC GRADE**

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 November 1976, after the draft finalized by the Photographic Chemicals and Related Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 Hydroxylamine sulphate is mainly used as a photographic developer.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for hydroxylamine sulphate suitable for processing of sensitized photographic materials.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of colourless crystals or white crystalline powder.

2.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of the appendix is given in col 4 of the table.

3. PACKING AND MARKING

3.1 Packing — The material shall be preferably packed in glass bottles with airtight stoppers or as agreed between the purchaser and the supplier. The containers shall be securely closed.

*Rules for rounding off numerical values (revised).

**TABLE 1 REQUIREMENTS FOR HYDROXYLAMINE
SULPHATE, PHOTOGRAPHIC GRADE**

(Clause 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Hydroxylamine sulphate content, percent by mass, <i>Min</i>	98.0	A-2
ii)	Residue on ignition, percent by mass, <i>Max</i>	0.05	A-3
iii)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	A-4
iv)	Iron (as Fe), percent by mass, <i>Max</i>	0.001	A-5
v)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.002	A-6
vi)	Free acidity	To pass test	A-7
vii)	Ammonium compounds (as NH_4), percent by mass, <i>Max</i>	0.1	A-8

3.2 Marking — Each container shall be legibly and indelibly marked with the following:

- a) Name of the material;
- b) Net mass of the material;
- c) Manufacturer's name and recognized trade-mark, if any; and
- d) Lot or batch number in code or otherwise.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING AND CRITERIA FOR CONFORMITY

4.1 Representative samples of the material shall be drawn and conformity of a lot to the standard determined as prescribed in Appendix B.

APPENDIX A

(Clause 2.2)

ANALYSIS OF HYDROXYLAMINE SULPHATE,
PHOTOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF HYDROXYLAMINE SULPHATE
CONTENT

A-2.1 Reagents

A-2.1.1 *Potassium Bromate Solution* — 0.1 N. Dissolve 2.785 g of potassium bromate, previously dried at 105°C, in water, dilute to 1 000 ml and mix well.

A-2.1.2 *Dilute Hydrochloric Acid* — approximately 2 N.

A-2.1.3 *Potassium Iodide* — solid.

A-2.1.4 *Standard Sodium Thiosulphate Solution* — 0.1 N.

A-2.1.5 *Starch Solution* — 1 percent (*m/v*), freshly prepared.

A-2.2 Procedure — Weigh accurately 0.3 g of the material in a beaker and dissolve it in 250 ml of water. Transfer 25 ml of the solution to a stoppered flask and to this add 50 ml of potassium bromate solution followed by 20 ml of hydrochloric acid. Allow the contents to stand for 1 hour and then add 3 g of potassium iodide. Titrate the liberated iodine with standard sodium thiosulphate solution using starch solution as indicator. Carry out a blank determination.

A-2.3 Calculation

$$\text{Hydroxylamine sulphate content, percent by mass} = \frac{13.68 (V_2 - V_1) \times N}{M}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution used in titration,

V_2 = volume in ml of standard sodium thiosulphate solution used in blank determination,

N = normality of standard sodium thiosulphate solution, and

M = mass in g of the material taken for the test.

*Specification for water, distilled quality (revised).

A-3. DETERMINATION OF RESIDUE ON IGNITION

A-3.1 Apparatus

A-3.1.1 *Platinum or Silica Basin*

A-3.1.2 Furnace — capable of maintaining temperature within $600 \pm 25^\circ\text{C}$.

A-3.2 Procedure — Slowly burn, in several portions, approximately 50 g of the material, weighed to the nearest 0.01 gram, in a tared platinum or silica basin and ignite in a furnace at $600 \pm 25^\circ\text{C}$. Cool in a desiccator and weigh. Retain the residue for determination of heavy metals (*see A-4*).

A-3.3 Calculation

$$\begin{array}{l} \text{Residue on ignition, percent} \\ \text{by mass} \end{array} = \frac{M}{M_1} \times 100$$

where

M = mass in g of the residue after ignition, and

M_1 = mass in g of the material taken for the test.

A-4. TEST FOR HEAVY METALS

A-4.0 Outline of the Method — The colour produced by heavy metals with hydrogen sulphide is compared with that produced with standard lead solution under identical conditions.

A-4.1 Apparatus

A-4.1.1 Nessler Cylinders — 50 ml capacity.

A-4.2 Reagents

A-4.2.1 Concentrated Nitric Acid — *See IS : 264-1976**.

A-4.2.2 Standard Lead Solution — Dissolve 1.60 g of lead nitrate $[\text{Pb}(\text{NO}_3)_2]$ in water, add 1 ml of concentrated nitric acid and make up the volume to 1 000 ml. When required for use, pipette out 10 ml of this solution and dilute to 1 000 ml with water. One millilitre of the diluted solution contains 0.01 mg of lead (as Pb).

A-4.2.3 Hydrogen Sulphide Solution — freshly prepared, saturated, aqueous.

*Specification for nitric acid (*second revision*).

A-4.3 Procedure — Dissolve 3.000 g of the sample preserved in A-3.2 in water and dilute to 30 ml in a graduated cylinder. Take 5 ml of this solution, add 4 ml of standard lead solution and dilute to 40 ml with water. Dilute the remaining sample solution to 40 ml with water. Add 10 ml of hydrogen sulphide solution to each of these solutions. Any colour produced in the sample solution shall not be greater than that produced in the treated standard lead solution.

A-5. TEST FOR IRON

A-5.1 Apparatus

A-5.1.1 Nessler Cylinders — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 *p*-Nitrophenol Indicator Solution — Dissolve 0.2 g of *p*-nitrophenol in hot water and dilute to 100 ml.

A-5.2.2 Dilute Ammonium Hydroxide — 1 : 9.

A-5.2.3 Dilute Hydrochloric Acid — 1 : 99.

A-5.2.4 Acetate Buffer Solution — Add 23 g of anhydrous sodium acetate to 58 ml of acetic acid (2 N) and dilute to 1 000 ml with water. Adjust the final pH of the solution to 5.0 ± 0.1 with acetic acid or sodium hydroxide solution (10 percent).

A-5.2.5 *o*-Phenanthroline Reagent — Thoroughly mix equal volumes of *o*-phenanthroline solution (0.1 percent aqueous), hydroxylamine hydrochloride solution (10 percent aqueous) and acetate buffer solution.

A-5.2.6 Standard Iron Solution — Dissolve 0.702 g of ammonium ferrous sulphate in about 100 ml of water containing 10 ml of dilute sulphuric acid (4 N) and dilute to 1 000 ml. Further dilute 100 ml of the solution to 1 000 ml. One millilitre of this diluted solution contains 0.01 mg of iron (as Fe).

A-5.3 Procedure — Dissolve 1.000 g of the material in 25 ml of water in a Nessler cylinder and add one drop of *p*-nitrophenol indicator. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid dropwise until the solution becomes colourless and then add 2 ml in excess. Add 5 ml of *o*-phenanthroline reagent, mix well and let stand for 10 minutes. Dilute to 50 ml and mix well. Simultaneously prepare a control in another Nessler cylinder, using 1 ml of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

A-5.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that in the control.

A-6. DETERMINATION OF CHLORIDE CONTENT

A-6.0 Outline of the Method — The opalescence produced by the standard chloride solution with silver nitrate solution is compared to that produced by the material under identical conditions.

A-6.1 Apparatus

A-6.1.1 Nessler Cylinders — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 Dilute Nitric Acid — approximately 5 N.

A-6.2.2 Silver Nitrate Solution — approximately 4 percent (*m/v*).

A-6.2.3 Standard Sodium Chloride Solution — Dissolve 0.1649 g of sodium chloride in water and make up the volume to 1000 ml. One millilitre of this solution contains 0.1 mg of chloride (as Cl). Further dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of chloride (as Cl).

A-6.3 Procedure — Dissolve 1.000 g of the material in water and transfer into a Nessler cylinder. Then add 6 ml of dilute nitric acid and 1 ml of silver nitrate solution. Make up the solution to 50 ml with water. Prepare a control in another Nessler cylinder, using 2 ml of standard chloride solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the opalescence produced in the two cylinders after 5 minutes.

A-6.4 The limit prescribed for chloride content shall be taken as not having been exceeded if the opalescence produced in the test with the material is not greater than that in the control.

A-7. TEST FOR FREE ACIDITY

A-7.1 Reagents

A-7.1.1 Rectified Spirit — 95 percent (*v/v*) (*see also* IS : 323-1959*).

A-7.1.2 Dimethyl Yellow Solution — Dissolve 0.2 g of dimethyl yellow in 100 ml of rectified spirit.

A-7.1.3 Standard Sodium Hydroxide Solution — 1 N.

*Specification for rectified spirit (*revised*).

A-7.2 Procedure — Dissolve 1·000 g of the material in 50 ml of water. Add a few drops of dimethyl yellow solution and titrate to full yellow colour with sodium hydroxide solution.

A-7.3 The material shall be taken to have passed the test if not more than 0·5 ml of 1 N sodium hydroxide solution is required.

A-8. DETERMINATION OF AMMONIUM COMPOUNDS

A-8.1 Apparatus

A-8.1.1 Beaker — 150 ml capacity.

A-8.1.2 Distilling Assembly — The assembly, as shown in Fig. 1, consists of a round-bottom flask *A* of 1 000 ml capacity fitted with a rubber stopper having two holes, through one of which passes one end of the connecting bulb tube *B* and through the other the end of the tap or separating funnel *F* which dips into the contents of the flask. The other end of the bulb tube is connected to the condenser *C*. The lower end of the condenser is attached by means of a rubber tube to a dip tube *D* which dips into a known quantity of acid (sulphuric or boric acid), contained in a beaker *E* of 500 ml capacity, to which 3 to 4 drops of indicator solution have been added.

A-8.2 Reagents

A-8.2.1 Sodium Hydroxide Solution — 10 percent.

A-8.2.2 Standard Sulphuric Acid Solution — 0·02 N.

A-8.2.3 Standard Sodium Hydroxide Solution — 0·02 N.

A-8.2.4 Formalin Solution

A-8.2.5 Nitrazine Yellow Indicator

A-8.3 Procedure — Weigh $5 \pm 0\cdot01$ g of the sample, transfer to a 150-ml beaker and dissolve in 75 ml of water. Adjust the pH of the solution to 11·0 with sodium hydroxide solution. Quantitatively transfer the adjusted sample solution, using very small amounts of water, to the round-bottom flask of the distillation assembly. Pipette 50·0 ml of standard sulphuric acid into a 250-ml conical flask. Place the conical flask under the delivery tube of the condenser, making sure that the delivery tube dips below the surface of the liquid. Start heating the distillation apparatus and distill at low heat until about 40 ml have distilled over. Remove the collection flask from the delivery tube, and add 8 drops of nitrazine yellow indicator and 1 ml of formalin solution previously neutralized to nitrazine yellow indicator. Titrate the solution with standard sodium hydroxide solution until the last trace of yellow colour disappears.

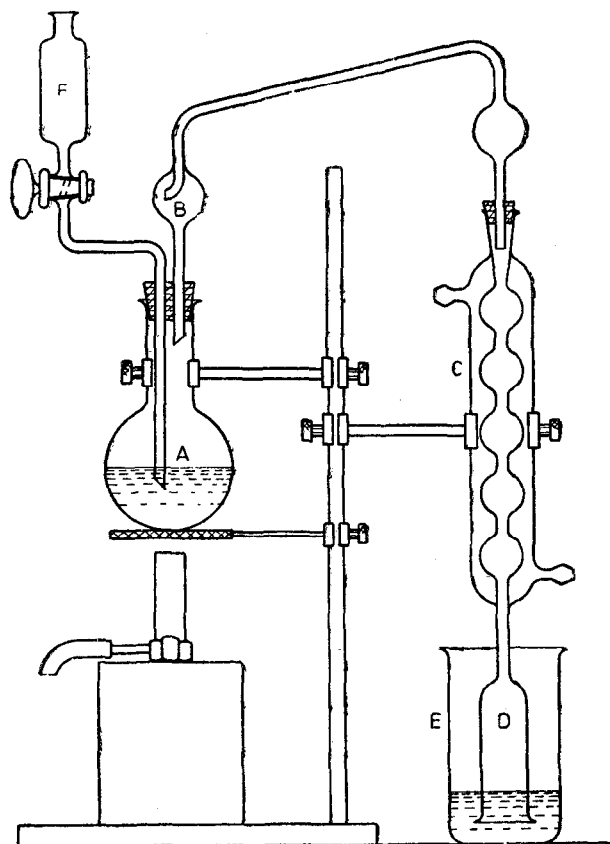


FIG. 1 DISTILLING ASSEMBLY FOR DETERMINATION OF NITROGEN

A-8.4 Calculation

$$\begin{array}{l} \text{Ammonium compounds (as } \text{NH}_4 \text{) ,} \\ \text{percent by mass} \end{array} = \frac{1.8 (V_1 N_1 - V_2 N_2)}{M}$$

where

V_1 = volume in ml of standard sulphuric acid solution added,

N_1 = normality of standard sulphuric acid solution,

V_2 = volume in ml of standard sodium hydroxide solution required in back titration,

N_2 = normality of standard sodium hydroxide solution, and

M = mass in g of the sample taken for the test.

A P P E N D I X B

(Clause 4.1)

SAMPLING OF HYDROXYLAMINE SULPHATE, PHOTOGRAPHIC GRADE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.3 The samples shall be placed in suitable, clean, dry and airtight glass containers on which the material has no action.

B-1.4 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.5 Each sample container shall be sealed airtight after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

B-2.3 The containers shall be selected at random from the lot. In order to ensure randomness of selection, procedures given in IS : 4905-1968* may be followed.

*Methods for random sampling.

TABLE 2 SCALE OF SAMPLING

(Clause B-2.2)

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
(1)	(2)
Up to 15	3
16 to 40	4
41 to 65	5
66 to 110	6
111 and above	7
	10

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.2** and **B-2.3**, a small representative portion of the material, about 100 g, shall be drawn with the help of a suitable sampling instrument.

B-3.2 Out of these portions, equal quantities of material shall be taken and mixed thoroughly to form a composite sample of mass about 150 g. The process of coning and quartering may be adopted to obtain the composite sample if the quantity of material mixed from different portions is large. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples shall be marked for the purchaser, another for the supplier and the third for use as referee samples.

B-3.4 All the individual and composite samples shall be transferred to separate sample containers. The containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed between the purchaser and the supplier, to be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of hydroxylamine sulphate content shall be performed on the individual samples.

B-4.2 Tests for all other characteristics shall be performed on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the test results for hydroxylamine sulphate content, the mean \bar{X} and range R of test results shall be computed (range being defined as the difference between the maximum and the minimum values of test results).

B-5.1.1 The lot shall be declared as conforming to the requirement for hydroxylamine sulphate content if the value of the expression $\bar{X} - 0.6 R$ is equal to or greater than 98.0.

B-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each characteristic shall satisfy the relevant requirement specified.

B-5.3 The lot shall be declared as conforming to the requirements of the specification if **B-5.1** and **B-5.2** are satisfied.

INDIAN STANDARDS

ON

PHOTOGRAPHIC CHEMICALS

IS:

- 246-1972 Sodium thiosulphate, crystalline (*third revision*)
- 247-1972 Sodium sulphite, anhydrous (*third revision*)
- 248-1971 Sodium bisulphite (sodium metabisulphite) (*second revision*)
- 332-1967 Chromium potassium sulphate (chrome alum) (*first revision*)
- 388-1972 Hydroquinone, photographic grade (*second revision*)
- 500-1972 Potassium metabisulphite, photographic grade (*second revision*)
- 557-1968 Sodium acetate, technical and photographic (*first revision*)
- 2211-1972 Anhydrous sodium thiosulphate, photographic grade (*first revision*)
- 2318-1974 Silver nitrate, photographic grade (*first revision*)
- 2797-1964 Potassium bromide
- 4173-1975 4-Methylaminophenol sulphate (*first revision*)
- 5379-1969 Ammonium thiosulphate, photographic grade
- 5380-1976 Sodium bromide, photographic grade (*first revision*)
- 5381-1969 Quantity packaging of sensitized photographic materials
- 5431-1969 Definition of motion-picture safety films
- 6139-1971 Sizes of photographic paper for general use
- 6212-1971 Method for the determination of residual thiosulphate in processed black and white photographic films and plates
- 8281-1976 Hydroxylamine sulphate, photographic grade

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